भारतीय मानक

Indian Standard

IS 17990 : 2022

हेक्सामिथाइल्डिसिलजेन — विशिष्टि

Hexamethyldisilazane — Specification

ICS 71.100.55

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भारतीय मानक ब्यूरो
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October 2022 **Price Group 4**

Plastics Sectional Committee, PCD 12

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Bis(trimethylsilyl)amine (also known as hexamethyldisilazane and HMDS) is an organosilicon compound with the molecular formula [(CH3)3Si]2NH. The molecule is a derivative of ammonia with trimethylsilyl groups in place of two hydrogen atoms. This colorless liquid is a reagent and a precursor to bases that are popular in organic synthesis and organometallic chemistry. Additionally, HMDS is also increasingly used as molecular precursor in chemical vapor deposition techniques to deposit silicon carbonitride thin films or coatings.

HMDS is synthesized by treatment of trimethylsilyl chloride with ammonia. For production of 1 kg of Hexamethyldisilazane, 1.5 Kg of trimethylsilyl chloride (TMCS) and 0.45 Kg of Ammonia (NH3) is used.

$2\ (CH3)_3SiCl + 3\ NH_3 \rightarrow [(CH3)_3Si]_2NH + 2\ NH_4Cl$

HMDS can cause toxic effects if inhaled or ingested/swallowed. Severe skin and eye burns may result from contact with HMDS.

Producing and using HMDS as a chemical intermediate may result in its release to the environment through various waste streams, which will further pollute the environment.

Based on the recommendations of Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizer, the Committee has decided to formulate Indian Standard on Hexamethyldisilazane (HMDS).

The composition of the Committee responsible for formulation of this standard is given in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2: 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

HEXAMETHYLDISILAZANE — SPECIFICATION

1 SCOPE

This Standard prescribes the requirements, methods of sampling and tests for Hexamethyldisilazane (HMDS) [also known as Bis(trimethylsilyl)amine].

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most editions of the standards indicated below:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 TERMINOLOGY

3.1 Organosilicon Compound — Compounds containing carbon-silicon bonds.

4 REQUIREMENTS

4.1 Description

The material shall be a clear colourless liquid.

4.2 The material shall comply with the requirements of Table 1 when tested with methods mentioned in the column 4 of the Table 1.

Table 1
(*Clause* 4.2)

Sl No.	Characteristics	Requirements	Method of Test, Refer to
(1)	(2)	(3)	(4)
i)	Solubility	Insoluble in water and soluble in acetone	Annex A
ii)	Specific Gravity, at 25ºC, *Max*	0.78	Annex B
iii)	Boiling Range, ºC	123 to 127	Annex C
iv)	Purity (By GC), Percent, *Min,*	99.0	Annex D
v)	Hexamethyldisiloxane (HMDO), percent, *Max*	0.5	-do-
vi)	Trimethylchlorosilane (TMCS), percent, *Max*	0.5	-do-

5 PACKAGING AND MARKING

5.1 Packing

The material shall be packed under nitrogen gas. The container shall be tightly closed in a dry and well-ventilated place and shall be kept away from heat and source of ignition. The material shall be packed in 160 Kgs epoxy coated MS drums, ISO tank.

5.2 Marking

5.2.1 The container shall be marked with the following:

a) Name of the material;
b) Physical state;
c) Net Weight, Tare Weight and Gross Weight;
d) Lot Number/Batch Number;
e) Manufacturer’s name and his recognized trademark, if any;
f) Safety Sign/Symbols; and
g) Any other statutory requirements.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

5.2.3 The containers shall be suitably marked in red letters as FLAMMABLE LIQUID and CORROSIVE LIQUID and shall be labelled as shown in Fig. 5 and Fig. 15 of IS 1260 (Part 1) respectively.

6 TEST METHODS

6.1 Tests shall be conducted according to the method of test referred in column 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

7 SAMPLING

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in 4 of IS 5299.

ANNEX A
SOLUBILITY

[*Table* 1, *Sl No.* i)]

A-1 PRINCIPLE

This method is used for the determination of solubility of HDMS material in water and acetone.

A-2 PROCEDURE

Take 2 test tubes with one having 5 ml water and other 5 ml acetone. Add 5 ml HDMS material in both the test tubes. Shake the test tubes and observe.

A-3 REPORT

Report the solubility of material in water and acetone.

ANNEX B
SPECIFIC GRAVITY

[*Table* 1, *Sl No.* ii]

B-1 PRINCIPLE

This method is used for the determination of specific gravity of HDMS material.

B-2 PROCEDURE

Take a 25 ml volumetric flask along with the stopper. Note down the weight of flask with stopper as W_1. Fill the flask with a HDMS material up to the mark. Note down the weight of the material with the flask as W_2.

B-3 CALCULATION

Weight of empty volumetric flask = W_1

Weight of volumetric flask with the material = W_2

Weight of sample $W_3 = (W_2 - W_1)$

$$\text{Specific gravity} = \frac{W3}{25}$$

B-4 REPORT

Report result to its nearest decimal value.

ANNEX C

BOILING RANGE

[*Table* 1, *Sl No.* iii]

C-1 PRINCIPLE

This method is used for the determination of boiling range of HDMS material.

C-2 PROCEDURE

Take the 100 ml of material and check the vapour temperature by using boiling point apparatus.

C-3 REPORT

Report results to its nearest decimal value.

ANNEX D

[Table 1, Sl No. iv), v) and vi)]

METHOD FOR DETERMINATION OF PURITY (HEXAMETHYLDISILAZANE), HEXAMETHYLDISILOXANE (HMDO) TRIMETHYLCHLOROSILANE (TMCS) BY GAS CHROMATOGRAPHY(GC)

D-1 OUTLINE OF THE METHOD

This procedure is used to determine the assay of HMDS, HMDO and TMCS. Capillary gas chromatography is used to determine the percentage of HMDS, HMDO and TMCS. Quantitative results are achieved through area percent.

D-2 APPARATUS

D-2.1 Any gas chromatograph equipped with Flame Ionization Detector (FID), a split-splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

Column	Capillary column of fused silica, with 6 percent Cyanopropylphenyl and 94 percent Dimethylpolysiloxabe phase with length 30.0 m, Inner diameter 0.53 mm and film thickness of 3.00 micron
Carrier Gas	Nitrogen
Detector	Flame Ionization Detector (FID)
Flow rate	20 ml/min
Column Oven temperature	50ºC
Injector temperature	170ºC
Detector temperature	250ºC
Injection Volume	0.2 µl
Split Ratio	1:10
Run Time	23.33 mins
Retention Time of HMDS	About 8.89 min

D-2.2 Column Oven Temperature Program

Rate (ºC/min)	*Temperature (ºC)*	*Hold Time (min)*
00	50.0	6.00
15.00	220.0	6.00

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H2, N2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

D-3 PROCEDURE OF ANALYSIS

Inject about 0.2 µl of standard and sample. Run the chromatogram and record the peak of the response.

D-4 CALCULATION

Report the retention time of major peak in minutes, and results on area percentage.

D-5 IDENTIFICATION

The retention time of the material shall match with the retention time of standard in the test of purity.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Plastics Sectional Committee, PCD 12

Organization	*Representative(s)*
Central Institute of Plastics Engineering & Technology (CIPET), Chennai	Prof (Dr) Shishir Sinha (***Chairperson***) Dr S. N. Yadav *(Alternate I)* Dr Smita Mohanty (*Alternate II*)
All India Plastics Manufacturers Association (AIPMA), New Delhi	Shri Deepak Ballani
Central Pollution Control Board, New Delhi	Ms. Divya Sinha Shri C. K. Dixit (*Alternate*)
Chemical and Petrochemical Manufactures Association (CPMA) New Delhi	Shri Mahinder Singh
Coca-Cola India Pvt. Ltd., Gurugram	Shri Virendra Landge Shri Rajendra Dobriyal (*Alternate*)
Consumer Association of India (CONCERT), Chennai	Shri G. Santhanarajan Shri M. R. Krishnan (*Alternate*)
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CSIR-Indian Institute of Toxicological Research (IITR), Lucknow	Dr V. P. Sharma Dr A. B. Pant (*Alternate*)
CSIR- National Chemical Laboratory (NCL), Pune	Dr P. R. Suresha Dr R. V. Gundloori (*Alternate*) Shrimati Sangeeta Hambir (*Alternate II*)
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Food Safety and Standards Authority of India (FSSAI), Delhi	Shri Chirag Gadi
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Indian Oil Corporation, R and D Centre, Faridabad	Shri G. S. Kapoor Shri Dhananjay Sahoo (*Alternate*)
Indian Pharmacopoeia Commission, Ghaziabad	Dr Jai Prakash Dr Manoj Kumar Pandey (*Alternate*)

Organization	*Representative(s)*
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Member Secretary
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This Indian Standard has been developed from Doc No.: PCD 12 (18965).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi